Date Issued: October 6, 1969

Document Y-1696

TID-4500

UNION CARBIDE CORPORATION
Nuclear Division

Y-12 PLANT

Contract W-7405-eng-26
With the US Atomic Energy Commission

HOMOGENIZATION EVALUATION OF A CAST URANIUM-
MOLYBDENUM ALLOY

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ABSTRACT

Studies have been made on the uranium-10 weight percent molybdenum alloy to determine the method and parameters of heat treatment for homogenization, the method and results of tensile testing the treated alloy, and the results of metallography performed on the material. In addition, metallography was performed on stock from the wrought alloy which demonstrated lack of homogeneity.

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SUMMARY

An evaluation was made to establish the necessity for, and the parameters conducive to, homogenization of cast uranium-10 weight percent molybdenum alloy. Specimens were cut from a depleted uranium alloy ring for the Super Kukla fast burst reactor and given heat treatments at 900, 1,000, and 1,090° C for varying periods of time. These specimens, plus some in the as-cast condition, were tensile tested and metallographically examined. In addition, metallography was performed on a specimen which had been hot rolled and vacuum heat treated. Tentative conclusions from the work are:

1. A homogenization treatment is necessary to develop 10 percent elongation in material containing 700 ppm carbon and having been cast by the standard ring procedure.
2. Nominal ultimate and yield strengths of 140,000 psi; elongations of 10 to 15 percent; and reductions in area of 12 to 30 percent can be achieved.
3. At temperatures and times of heat treatment where grain growth occurred, strength was reduced by approximately 10 percent and elongations were reduced to 5 to 7 percent.
4. Metallography indicated definite evidence of coring in the as-cast alloy.
5. Heat treatment at 900° C for 24 hours, 1,000° C for 12 hours, and 1,090° C for six hours all homogenized the material to a degree better than the sensitivity of the metallographic etching procedure.
6. Heat treatment of the alloy at 1,090° C for six hours or more can cause massive grain growth.
7. Homogenizing the alloy prior to working is definitely desirable to produce a chemically homogeneous wrought product.

INTRODUCTION

The Super Kukla fast burst reactor utilized components made of cast uranium-10 weight percent molybdenum alloy. Specifications required that these components meet the minimum tensile requirements (110,000 psi yield strength and 10 percent elongation).

A question arose concerning the possible necessity of performing a homogenizing treatment on the alloy in order to meet the requirements. Available phase diagrams indicated that the 10 percent alloy composition could segregate during solidification with cored gradients up to a range of 2 to 15 weight percent molybdenum.

This report includes a brief description of the alloy that was tested, the method and parameters of heat treatments for homogenization, the method and results of tensile testing, and the methods and some results of the metallography performed on the heat-treated alloy.

EVALUATION OF THE URANIUM-MOLYBDENUM ALLOY

SPECIMEN STOCK AND HEAT TREATMENT

The samples were taken from a ring of a development casting having nominal dimensions of 30 inches outside diameter, 18 inches inside diameter, and approximately 2 1/4 inches high. Chemical analysis data of the ring are reported in Table 1. The 1/2 by 1/2 by 4-inch specimen blanks were removed from scattered locations in the test ring—three specimens per location through the ring thickness. Each ring was identified only by its relative location in the thickness of the ring: Specimen 1 representing the bottom of the cast ring, Specimen 2 the approximate middle, and Specimen 3 the approximate top after all machining had been performed to remove casting flaws.

Table 1
CHEMICAL ANALYSIS DATA FOR TEST RING

Element	Concentration (ppm)	Element	Concentration (ppm)
Al	6	Mg	2
As	< 10(1)	Mn	100
Au	< 1	Mo	10.08(2)
B	0.2	Na	6
Ba	< 2	Nb	< 4
Be	8	Ni	60
Bi	< 1	P	< 100
C	728	Pb	2
Ca	< 10	Pd	< 1
Cd	< 0.1	Rb	< 20
Co	< 1	Sb	< 2
Cr	< 2	Si	50
Cs	< 100	Sn	1
Cu	40	Sr	< 20
Fe	350	Ti	< 4
Ga	< 1	Tl	< 4
Ge	0.4	V	< 1
Hf	< 100	W	< 100
In	< 2	Zn	< 10
K	< 6	Zr	< 20
Li	< 0.2		

(1) < indicates the limit of sensitivity of the analytical method used.

(2) Weight percent.

Specimens, randomly selected in groups of three, were heat treated with varying time and temperature parameters. The heat treatments were performed in a resistance-heated vacuum furnace at a sustained vacuum of less than 10^{-4} mm (Hg) and a maximum temperature fluctuation of $\pm 10^{\circ}$ C at temperature. Heating was controlled at a rate of approximately $1,200^{\circ}$ C per hour; the furnace was cooled at a controlled rate of 200° C per hour.

TENSILE TESTING

After heat treatment, the specimen blanks were machined into shoulder-end "252" tensile specimens. Figure 1 gives the dimensions of the specimen. Tensile testing was performed at a strain rate of 0.05 inch per inch per minute from start to failure. A typical stress-strain graph is presented in Figure 2. All of the specimen tensile data and their respective heat-treatment history are listed in Table 2.

Figure 1. SHOULDER-END TENSILE SPECIMEN.

Figure 2. TYPICAL TENSILE STRESS-STRAIN GRAPH OF A HOMOGENIZED URANIUM-MOLYBDENUM ALLOY SPECIMEN.

Table 2
TENSILE DATA FOR A URANIUM-MOLYBDENUM ALLOY HOMOGENIZATION EVALUATION

Cycle	Specimen	Ultimate and Yield Strength (psi $\times 10^{-3}$)	Elongation(1) (%)	Reduction in Area (%)	Comments	Cycle Condition
1	2	140.0	1.0	0.8		As Cast
	2A	143.1	2.5	-		
	3	137.5	0.5	-	Apparent casting flaw at fracture.	
2	1A	136.5	11.0	21.1		900° C - 24 hours
	2	139.7	10.0	14.5		
	1	143.2	13.5	24.2		
3	1	141.5	11.0	28.7		900° C - 48 hours
	1A	141.5	9.5	13.1		
	2	140.9	12	19.3		
4	3	139.5	8.0	11.6		1,000° C - 24 hours
	1	142.1	15.0	26.5		
	2	141.5	13.5	20.4		
5	2	142.5	14.5	31.4		1,000° C - 12 hours
	3	140.9	0.5	2.8		
	2A	140.3	11.0	15.4		
6	2	140.7	15	27.4		1,090° C - 24 hours
	3	126.0	5	11.3		
	1	128.3	6.5	19.0	Large shear lip at fracture.	
7	1	127.3	6	23.2	Large shear lip at fracture.	1,090° C - 6 hours
	2	141.3	14.5	28.7		
	3	135.6	11	21.8	Moderate shear lip at fracture.	
8	2	140.4	15.0	27.3		1,090° C - 6 hours
	3	138.4	5.0	4.4		
	1	126.5	7.0	29.3	Large shear lip at fracture.	
9	3	108.9	0	0	Apparent casting flaw at fracture.	500° C - 24 hours
	3A	142.7	11.0	20.7		plus
	1	141.7	15.5	28.7		1,000° C - 12 hours
10	3A	138.9	7.5	10.5		500° C - 48 hours
	1	142.7	13.0	24.9		plus
	3	139.3	5.0	4.4		1,000° C - 12 hours

(1) In one inch.

METALLOGRAPHY OF THE CAST MATERIAL

Metallography was performed on specimens of the as-cast alloy and specimens that were subjected to each of the heat-treat conditions. Figure 3 is a photomicrograph of Specimen 2-2 which received 24 hours of heat treatment at 900° C. Most previous homogenization treatments of uranium-10 weight percent molybdenum have been performed with these parameters. Figure 4 is a view of Specimen 5-2 which received 12 hours of heat treatment at 1,000° C. Little difference can be noted between the two structures other than a possible slightly larger grain size in Specimen 5-2 which received the higher temperature heat treatment. The major change in structure noted in all of the heat-treated specimens is indicated in Figure 5. It shows the structure of

5330-2-2

Figure 3. HEAT-TREATED STRUCTURE OF CAST URANIUM-MOLYB-DENUM ALLOY. (Heat Treated for 24 Hours at 900°C ; Bright Field Illumination; 100X)

5330-5-2

Figure 4. HEAT-TREATED STRUCTURE OF CAST URANIUM-MOLYB-DENUM ALLOY. (Heat Treated for 12 Hours at $1,000^{\circ}\text{C}$; Bright Field Illumination; 100X)

(a) At 7.5X

5330-6-2

(b) At 100X

5330-6-2

Figure 5. HEAT-TREATED STRUCTURE OF CAST URANIUM-MOLYBDENUM ALLOY. (Heat Treated for 24 Hours at 1,090° C)

Specimen 6-2 which received 24 hours of heat treatment at 1,090° C. Apparently, isolated grains had experienced a considerable amount of growth. Figure 5(a) is a photomacrograph of the 1/2-inch-diameter head of the tensile specimen showing two extremely large grains separated by a zone of as-cast grain structure. Figure 5(b) shows the boundary between one of the larger grains and the adjacent smaller-grain structure at 100X.

Secondary results were experienced when metallography was performed on the as-cast material. When the same electroetching technique was used, tinting resulted at, and adjacent to, most of the cast grain boundaries (Figure 6). The etching method was sensitive to chemical inhomogeneity within the alloy. The tinted areas distinguish the zones which would, ordinarily, be expected to be molybdenum deficient, if a normal mode of coring solidification had taken place. However, when the homogenized specimens were electroetched, no tinting was noted; and a total of 40 seconds of etching time was required to resolve the grain boundaries satisfactorily. With the as-cast specimen, five seconds of etch started a slight shadow of tint, as Figure 6(a) indicates; and, in ten seconds, a heavy tint developed in areas adjacent to the grain boundaries. First sight of the photomicrograph, Figure 6(b), showing the heavy tint gives the impression of two distinct major phases with a rather distinct boundary. But, in working with the specimen this idea did not appear to be true, as may be noted in Figure 6(a) which contains only a shadow tint with no apparent phase distinction or boundary. It was concluded that the boundary effect on the heavily tinted specimen was a chemical phenomenon of the etching rather than a dual-phase characteristic of the material.

METALLOGRAPHY OF THE HOT-ROLLED AND HEAT-TREATED MATERIAL

The etching procedure was also used on a specimen from a wrought uranium-10 weight percent molybdenum alloy tensile bar which was heavily attacked and grooved during electrolytic cleaning in preparation for nickel plating (Figure 7). The specimen was sectioned from the bar to give a cross section of the grooved surface. Electroetching by the previously mentioned metallographic technique developed tinted bands on the structure, some of which terminated at the roots of the surface grooves, as shown in Figure 8. These tinted areas, apparent evidence of chemical inhomogeneity, contained a major portion of the larger inclusions generally referred to as carbides. This result is consistent with previous experience on electroetched, as-cast specimens where the molybdenum depleted areas and the major portion of the carbide inclusions are jointly associated with the same zones—the cast grain boundaries.

The history of the wrought alloy from which this specimen was machined follows: Stock was cast, cropped, and cleaned to give a 28 1/2 by 22 by 5 1/2-inch rolling

(a) Five-Second Electroetch

5330-1-2

(b) Ten-Second Electroetch

5330-1-2

Figure 6. AS-CAST URANIUM-MOLYBDENUM ALLOY. (100X)

Figure 7. ELECTROCHEMICALLY GROOVED SPECIMEN OF WROUGHT URANIUM-MOLYBDENUM ALLOY.

billet. The billet was given a preheat of two hours at $1,000 - 1,025^{\circ}\text{C}$, then hot rolled to a final thickness of $5/8$ inch. Two reheats were performed, one before an intermediate shearing operation and the second before the final reduction from $1\ 3/8$ inches to $5/8$ inch in thickness. In all, the billet experienced no more than $2\ 3/4$ to $3\ 1/4$ hours in the 800 to $1,000^{\circ}\text{C}$ temperature range. After rolling, the $5/8$ -inch-thick plate was vacuum heat treated at 760°C for eight hours for the dual purpose of annealing and outgassing the major portion of the hydrogen that might have been picked up during the hot working. The specimen was then machined and electroetched in preparation for nickel plating. Chemical analysis data of the wrought alloy are listed in Table 3.

REVIEW OF RESULTS

Tensile test results definitely indicate that homogenization was necessary to achieve elongations of 10 percent or better. The balance of the specimens having less than 10 percent elongation, except one which had a marginal $9\ 1/2$ percent, fell into two categories—they were either specimens removed from

(a) At 20X

(b) At 100X

(c) At 250X

Figure 8. MACRO CROSS-SECTION VIEWS OF AN ELECTROCHEMICALLY GROOVED TENSILE SPECIMEN.

Table 3
CHEMICAL ANALYSIS DATA FOR WROUGHT URANIUM-
MOLYBDENUM ALLOY PLATE

Element	Concentration (ppm)	Element	Concentration (ppm)
Al	8	Li	0.5
As	< 10(1)	Mg	5
Au	< 1	Mn	10
B	< 0.1	Mo	9.51(2)
Ba	< 2	Na	10
Be	< 0.01	Nb	< 4
Bi	< 100	Ni	20
C	490	P	< 100
Ca	< 10	Pb	5
Cd	< 0.1	Pd	< 1
Co	< 1	Rb	< 20
Cr	2	Sb	< 2
Cs	< 100	Si	20
Cu	15	Sn	< 1
Fe	80	Sr	< 20
Ga	< 1	Ti	< 4
Ge	0.4	Tl	< 4
Hf	< 100	V	< 1
In	< 2	W	< 100
K	< 6	Zn	< 10

(1) < indicates the limit of sensitivity of the analytical method used.

(2) Weight percent.

stock near the top of the casting or they had received the highest temperature (1,090° C) heat treatment. The low-elongation specimens which were heat treated at the highest temperature had relatively large shear lips at the fracture. This observation was not consistent with the balance of the specimens or previous experience on tensile fractures of uranium-10 weight percent molybdenum alloy. These same specimens also exhibited a lower strength of approximately 10 percent.

Metallography showed definite evidence of coring in the as-cast structure. All the heat-treated specimens appeared to be homogeneous within the limits of sensitivity of the metallographic etching technique. Slight differences in grain size were found in the specimens heat treated at the lower temperatures, but these differences did not deviate much from the range or size that can be expected from the top to the bottom of the casting. The 1,090° C heat-treated specimens did exhibit isolated instances of massive grain growth which explains the rather unusual fracture mode of some of these specimens.

The wrought specimen showed metallographic evidence of chemical nonhomogeneity. It was originally thought that the hot working and continual or repeated recrystallization might speed up the diffusion process to give a homogeneous

material after the final heat treatment. However, a thorough homogenization heat treatment was necessary before, or at, an intermediate stage of rolling to insure chemical homogeneity of the alloy.

It was shown that massive grain size, due to heat treatment, was detrimental to the strength and elongation capabilities of the alloy. The material used contained approximately 700 ppm carbon. Examination of lower-carbon-content material should be performed to establish the effect of carbon on strength and the allowable heat-treatment temperature limit to minimize gross grain growth. With high purity, this limit might be lowered.